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Microstructure characterization and optical properties of sapphire after helium ion implantation

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ABSTRACT

The (0001) sapphire samples are irradiated with 60 keV helium ions at the fluences of 5×10^{16}, $1 \times 10^{17}$and 5×10^{17} ions/cm^2 at room temperature. After implantation, two broad absorption bands at 320–460 and 480–700 nm are observed and their intensities increase with the increasing ion fluence. The grazing incidence X-ray diffraction results indicate that the {0001} diffraction peaks of sapphire decrease and broaden due to the disorientation of the generated crystallites after ion irradiation. The microstructure evolution is examined by the scanning and transmission electron microscopes. The surface becomes rough because of the aggregation of helium bubbles and migration towards the surface. There is a lattice expansion up to ~4.5% in the implanted area and the lattice distortion measured from dispersion of (110) diffraction is ~4.6°. Such strain of crystal lattice is rather large and leads to contrast fluctuation at scale of 1–2 nm (the bubble size). The laser induced damage threshold (LIDT) is investigated to understand the effect of helium ion beam irradiation on the laser damage resistance of sapphire components and the results show that the LIDT decreases from 5.4 to 2.5 J/cm^2 due to the absorptive color centers, helium bubbles and defects induced by helium ion implantation. The laser damage morphologies of samples before and after ion implantation are also presented.

Single crystal α-Al_2O_3 (sapphire), as a wide band gap (8.8 eV) insulator, has been often used in microelectronics for photoelectronic devices or as window materials [1,2]. Actually, alumina (Al_2O_3), either in the polycrystalline ceramic form or as sapphire, is one of the insulating candidate materials to be used in diagnostic systems for the International Thermonuclear Experimental Reactor (ITER) [3]. In addition, in the high power solid-state laser driver for inertial confinement fusion (ICF), sapphire is also a good optical material. During nuclear fusion, besides laser beam, sapphire will be irradiated by other beams or rays, e.g., electron beam, ion beam, neutron beam, X and γ-rays [4]. Therefore, the optical material will be inevitably damaged and its structural and optical properties as well as laser damage resistance will change. The alpha particle is one of the important ion beams. In particular, the high helium production rate of about 5000 appm/year in the bulk by nuclear transmutation with consequences for electrical and mechanical behavior calls for a detailed understanding of the effect of helium on material properties [5].

In the recent decades, the development of nuclear reactor engineering has stimulated many investigations on the radiation damage mechanisms of sapphire produced by irradiation with energetic electrons, ions, neutrons or photons [6]. The interest in the behavior of helium in Al_2O_3 arises from two applications: (1) Al_2O_3 is a candidate host matrix for the transmutation of actinides generated in nuclear reactor fuel. Alpha decay of actinides introduces helium atoms into the Al_2O_3 matrix that interact with radiation damage defects. (2) Al_2O_3 is a popular host material for the generation of linear and non-liner optical properties by introducing metal or semiconductor nanoclusters [7]. Ion irradiation of inert gas atoms such as He can improve the understanding of defect evolution in the host material. Various studies have been performed on the He ion irradiated Al_2O_3, including microstructure evolution, He bubble formation, electrical degradation, and luminescence [3,5–9]. However, up to now, there has no report about the effect of ion beam irradiation on the laser damage resistance of sapphire. In this work, the optical absorption, microstructure evolution

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under irradiation, bubble formation, and laser induced damage (LID) of helium-ion-implanted sapphire are investigated.

1. Materials and methods

Optical polished (0001) sapphire were irradiated with 60 keV He^+ ions at the nominal fluences of 5×10^{16}, 1×10^{17} and 5×10^{17} ions/cm^2 in a vacuum chamber of 2.0×10^{-3} Pa. The ion flux was controlled at 1.1×10^{14} ion/cm^2 s. The incident angle between the main crystallographic direction of sapphire and the ion beam is about 7° to avoid the channeling effects during the implantation process. The samples were kept at room temperature with circulating cooling water during ion implantation.

The projected range of He ions and sputtering yield induced by He ion implantation are calculated by SRIM 2008 code [10], which is used to understand the helium ion distribution and surface sputtering. For calculation, the density of sapphire is 3.98 g/cm^3 and displacement energy of 40 eV is used for Al and O atoms [11]. The projected depth is about 288 nm and the sputtering yield is 0.033 atoms/ion for O and 0.016 atoms/ion for Al, respectively. Before and after ion implantation, the crystals were monitored using optical absorption spectroscopy. The optical absorption spectra were measured by a SHIMADZU UV-2550 spectrophotometer at room temperature, with a deuterium lamp for UV and a tungsten halogen lamp for the visible region. The wavelength used in the experiment ranged from 200 to 800 nm. The grazing incidence X-ray diffraction (GIXRD) spectra of samples were collected using a Philips X'Pert Pro MPD type X-ray diffractometer with a Cu Kα line of 1.54056 Å. The incident angle was 0.3° and the voltage and current were 60 kV and 60 mA, respectively. A FEI Helios Nanolab 650 scanning electron microscopy (SEM) operating at 30 kV with current of 0.4 nA was used to obtained the surface morphologies of the samples before and after He ion implantation. A JEOL2010F STEM/TEM (scanning transmission electron microscopy/transmission electron microscopy) analytical electron microscope operating at 200 keV was used for bright-field (BF), high-angle annular dark-field (HAADF), selected area electron diffraction (SAED) and high-resolution transmission electron microscopy (HRTEM) imaging to understand the microstructure evolution after ion implantation. The TEM specimen is prepared by an advanced focus ion beam (FIB) lift-out method, which has been published elsewhere.

In order to understand the laser damage resistance capability of optical materials used for laser facilities, the laser induced damage threshold is often tested. The laser induced damage threshold were conducted by using a single mode Nd:YAG laser operated at 355 nm with pulse width of 4.6 ns. The schematic diagram of experimental set-up for damage test is shown in Fig. 1. The laser beam was a spatial near-Gaussian distribution with beam area of 1 mm^2 at $1/e^2$. The beam areas were observed by a science CCD camera to monitor the initial damage in several microns in the surface of sapphire samples. The laser fluence fluctuates less than 5%. An EMP 1000 energy meter was used to collect the energy data of each shot. A Nikon LV100D optical microscope was utilized to observe the laser damage morphology of samples.

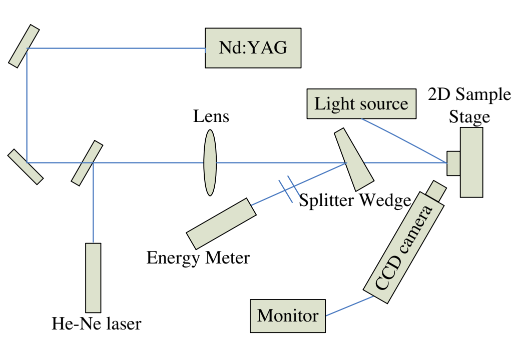

Fig. 1. Schematic diagram of experimental set-up for LIDT test.

2. Results and discussion

2.1. Optical absorption

The optical absorption spectra of α-Al_2O_3 single crystals after He ion implantation at nominal fluences of 5×10^{16}, 1×10^{17} and 5×10^{17} ions/cm^2 are shown in Fig. 2. In all spectra, three absorption bands at 205, 225 and 255 nm are observed, which are due to the intrinsic defects, negative ion vacancy, in the crystalline α-Al_2O_3. Furthermore, the strong UV absorption peak at 205 nm is ascribed to F centers and the absorption bands at 225 and 255 nm are attributed to F^+ centers, which are well-known basic electronic point defects in α-Al_2O_3, i.e., single oxygen vacancy trapping two or one electrons [12,13].

It is clearly revealed that there is similar tendency for all implanted samples. After ion implantation, two broad absorption bands at 320–460 and 480–700 nm appear and their intensities increase with the increasing ion fluence. Therefore, it can be deduced that the same types of radiation induced defects appeared in the samples implanted with different ion fluences. However, the concentration of defects increases with the increasing implantation fluence, so the intensities of the absorption bands increase accordingly. The previous studies indicated that the aggregated oxygen vacancies in the form of dimer F_2^+ center (two nearest or next-nearest-neighbor oxygen vacancies trapping three electrons) showing absorption at 358 nm (the superscript denotes the effective charge of the defects with respect to the ideal lattice) [14–16]. Therefore, the absorption band from 320 to 460 nm probably arises from the F_2^+ color centers. There has no report on the absorption band in the range between 480 and 700 nm for the pure α-Al_2O_3 single crystals. However, radiation and doping will introduced some new color centers. Abdukadyrova reported the absorption bands at 460, 570, 620, and 780 nm in the neutron irradiated sapphire. They calculated the activation energies and the nature of the defects involved: the 460-, 570-, 620-, and 780-nm color centers, were identified as F_2^{2+}, F_3, $[Al–O]^{3-}$, and $(Al–O^-)$ centers [17]. Sanyal et al. also reported the absorption bands near 525 and 620 nm in the C and Mg co-doped Al_2O_3 crystals [16]. They ascribes the absorption to the aggregated defects, in the form of impurity-

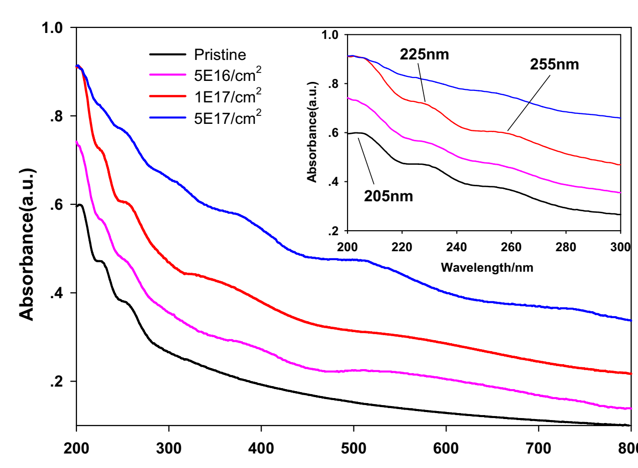

Fig. 2. Optical absorption spectra of sapphire before and after He ion implantation at nominal fluences of 5×10^{16}, 1×10^{17} and 5×10^{17} ions/cm^2.